# International Energy Agency (IEA IA-AMT) International Characterization Methods

# (Agreement ID:26462)


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PM064




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# Overview

## Timeline

- Project start date: Q4 FY13
- Project end date: FY15
- Percent complete: 25%

## Budget

- Total project funding
  - DOE share: $225k
- Funding received in FY13: $50k
- Funding for FY14: $175k

## Barriers

### Barriers addressed

- Changing internal combustion engine combustion regimes
- Long lead times for materials commercialization
- Cost

## Partners

- **USA: GMZ (G. Joshi); Clemson (T. Tritt); Marlow (J. Sharp); GM R&D (J. Salvador); Army Research Lab (P. Taylor), NIST (J. Martin)**
- **China: SICCAS (S.Q. Bai, L. Chen)**
- **Canada: CANMET(Y.C. Tseng); University of Waterloo (H. Kleinke);**
- **Germany: Fraunhofer IPM (J. König)**
- **United Kingdom: NPL (A. Cuenat)**
- **South Korea: KERI (M.H. Lee)**
- **Project lead: ORNL (H. Wang)**



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# Relevance/Objectives

## Objectives:

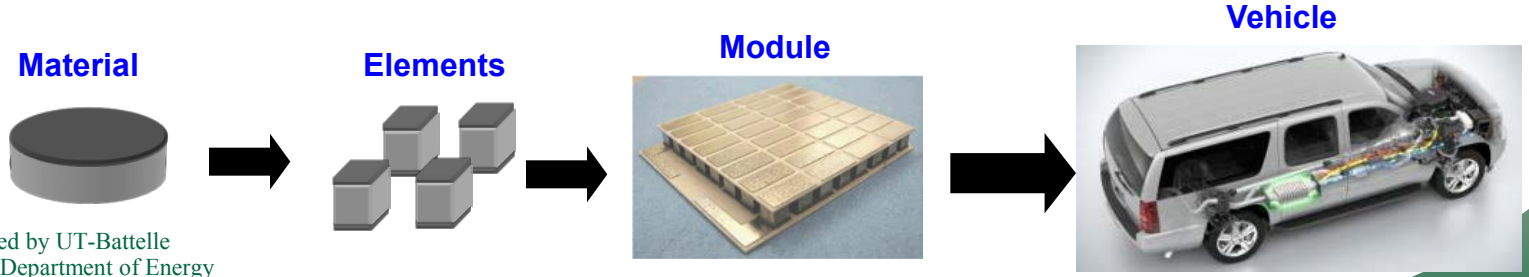
- **Materials:** Conduct international round-robin studies on materials characterization methods and develop standard testing procedures for vehicle energy conversion materials.
- **Devices:** Organize collaborative international studies on *energy conversion devices* for vehicles and develop standard testing methods and procedures.

## Addresses Targets:

- Standardization is the key for the commercialization of thermoelectric materials to recover engine waste heat.
- Thermoelectrics are experiencing the transition from materials R&D to manufacturing. Developing international standards can reduce the lead time to mass production.

## Uniqueness and Impact

- Material characterization and efficiency evaluation are important to realize their full potentials in fuel economy improvement and production scale vehicles.
- The main challenges are the lack of international standards in characterization of the materials and lack of standard testing procedures for device testing.
- This IEA project seeks broad international collaboration and promote reliable material measurements and device testing.



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# Milestones and Deliverbles

| Date | Quarter | Milestone | Status |
|---|---|---|---|
| 12/31/2013 | Q1 | Complete international round-robin on half heusler and report to IEA-AMT at the ExCo meeting in Hong Kong (December 2-3, 2013) | Completed |
| 3/31/2014 | Q2 | Conclude round-robin study and final report to IEA. Start international survey on efficiency testing of thermoelectric modules. | Completed |
| 4/30/2014 | Q3 | Deliverable: Submission of presentation for VTP annual Merit Review | On Track |
| 6/30/2014 | Q3 | Complete international survey on module efficiency testing. Report to IEA-AMT ExCo (location TBD). | On Track |
| 10/30/2014 | Q4 | Deliverable: Submission of Annual Progress Report to VTP Manager for publication | |
| 12/30/2014 | Q5 | Conduct international collaborative study on the measurement reliability of low-dimensional thermoelectrics | |
| 12/30/2014 | Q5 | Go/No-Go decision to continue research into next year. | |



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# Approach

***Materials level:* International round-robin #3 on half-heusler material**

- GMZ Energy volunteered their n-type half-heusler materials for the round-robin effort
- Materials processed and machined at GMZ Energy, n-type composition: $Hf_{0.5}Ti_{0.25}Zr_{0.25}NiSn_{0.99}Sb_{0.01}$
- Round-robin schedule: GMZ->Germany->China->US (ORNL, Clemson, GM R&D)->UK (NPL)-> Canada (CANMET) -> NIST->ARL->Marlow-> Korea (KERI)
- RT-500ºC to cover automotive exhaust temperature range
- Report to IEA-AMT and publish the results

***Materials level:* International collaborative study on the measurement reliability of low-dimensional thermoelectrics**

- Properly assess the reliability of transport data and figure of merit (ZT)
- Prepare the survey and report April-September 2014

***Device level:* Conduct and complete survey of module efficiency testing**

- Study current testing methods in collaboration with Marlow Industries, GM Global R&D, AIST (Japan) and Fraunhofer IPM (Germany)



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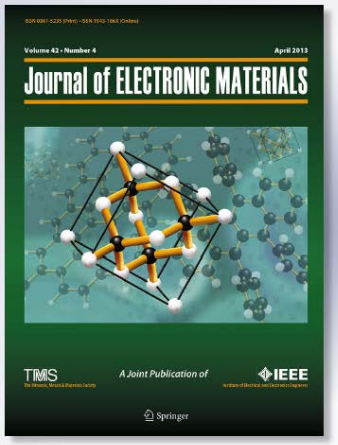
# Technical Accomplishments – 1 of 12

## Two IEA-AMT Annex VIII Publications in 2013

*Transport Properties of Bulk Thermoelectrics—An International Round-Robin Study, Part I: Seebeck Coefficient and Electrical Resistivity*

Hsin Wang, Wallace D. Porter, Harald Böttner, Jan König, Lidong Chen, Shengqiang Bai, Terry M. Tritt, Alex Mayolet, Jayantha Senawiratne, et al.

Journal of Electronic Materials
ISSN 0361-5235
Volume 42
Number 4
Journal of Elec Materi (2013) 42:654-664
DOI 10.1007/s11664-012-2396-8




Springer

*Transport Properties of Bulk Thermoelectrics: An International Round-Robin Study, Part II: Thermal Diffusivity, Specific Heat, and Thermal Conductivity*

Hsin Wang, Wallace D. Porter, Harald Böttner, Jan König, Lidong Chen, Shengqiang Bai, Terry M. Tritt, Alex Mayolet, Jayantha Senawiratne, et al.

Journal of Electronic Materials
ISSN 0361-5235
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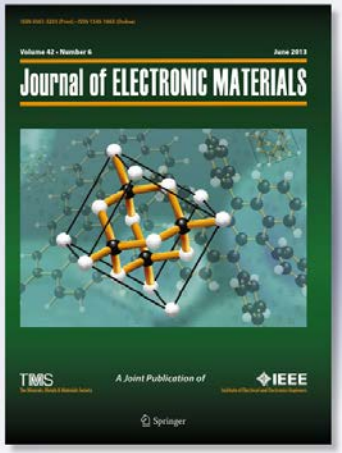
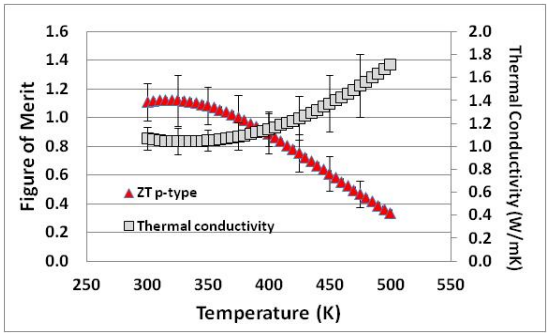
42:1073-1084
DOI 10.1007/s11664-013-2516-0




Springer

Preliminary round-robin studies produced two articles addressing transport properties measurement issue in bulk materials

### Reality Check on Testing: Error Bars in ZT




**+11% to +23% Scatter in ZT!**

Figure of Merit

$ZT = \sigma s^2 T/k$

$k = \alpha D C_p$

$\sigma$: Electrical Conductivity
S: Seebeck Coefficient
k: Thermal Conductivity
$\alpha$: Thermal Diffusivity
D: Density
Cp: Specific Heat



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# Technical Accomplishments – 2 of 12

## IEA-AMT Annex VIII RR3 Tests

**Notes:**
Compare to previous round-robin studies: A wider variety of test instruments were used in RR3

Several labs could not perform all tests and several labs obtained ZT using a different method

| | Lab | Diffusivity | Cp | Seebeck/Resistivity |
|---|---|---|---|---|
| 1 | GMZ | Netzsch 457 | Netzsch 404 | ULVAC ZEM-3 |
| 2 | ORNL | Netzsch 457 | Netzsch 404 | ULVAC ZEM-3 |
| 3 | Fraunhoffer | Netzsch 457 | Netzsch 404 | IPM-SRX-900K/ZEM-3 |
| 4 | SICAIC | Netzsch 457 | Netzsch 404 | ULVAC ZEM-3 |
| 5 | GM R&D | TA Xplatform | Netzsch 404 | Linseis LSR3 |
| 6 | NPL | Netzsch 427 | TA Q2000 | Home made system |
| 7 | CANMET | Anter/TA | Netzsch 404 | ULVAC ZEM-3 |
| 8 | Marlow | Harman* | Harman* | Harman |
| 9 | ARL | Steady-state* Isotherm | Steady-state* Isotherm | Steady-state Isotherm |
| 10 | NIST | N/A | N/A | Home-made system |
| 11 | KERI | Netzsch 457 | Netzsch 404 | ULVAC ZEM-3 |

* direct thermal conductivity measurement



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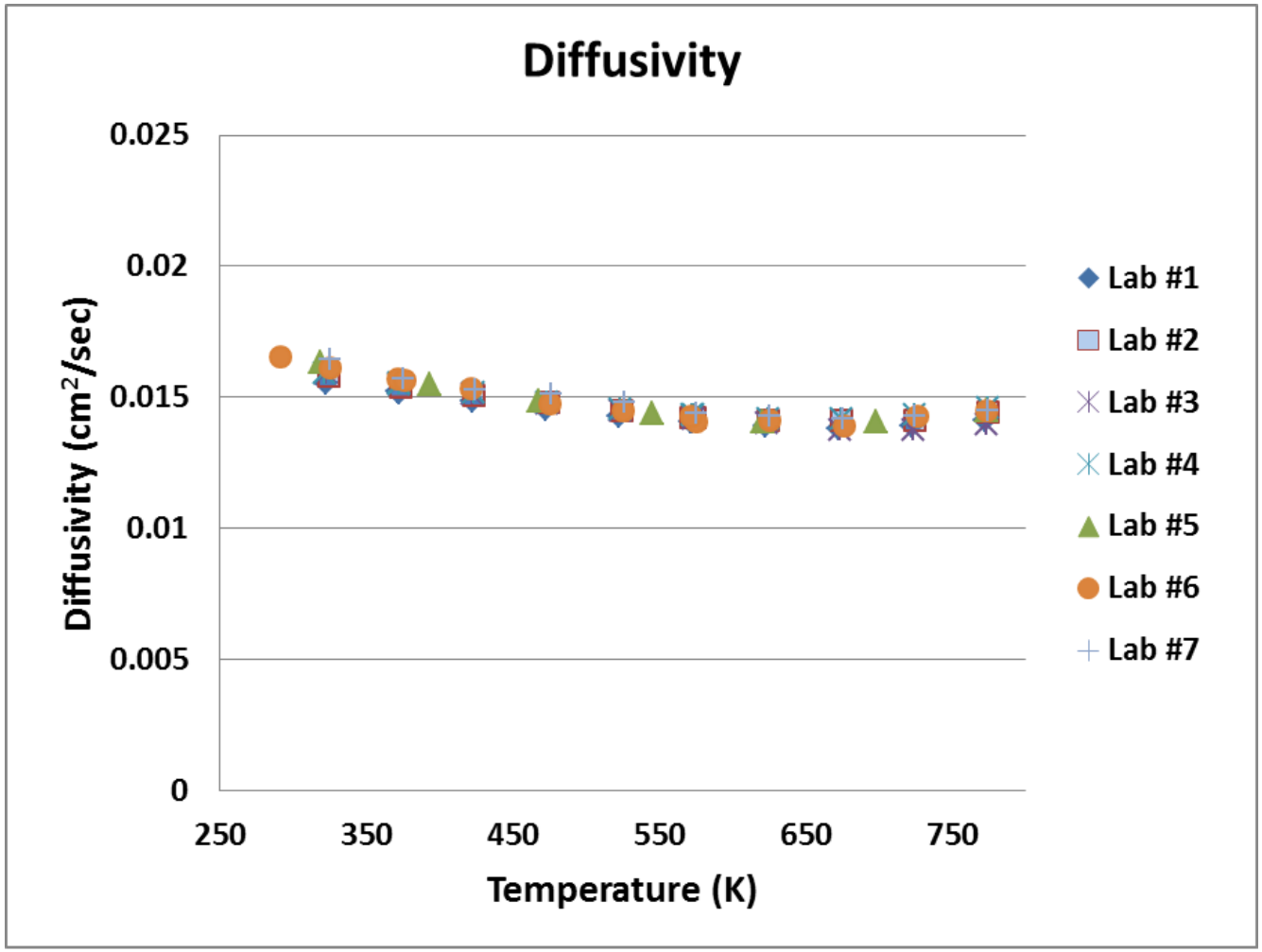
# Technical Accomplishments – 3 of 12

## IEA-AMT RR3: Diffusivity




**Very good agreement among participating labs**



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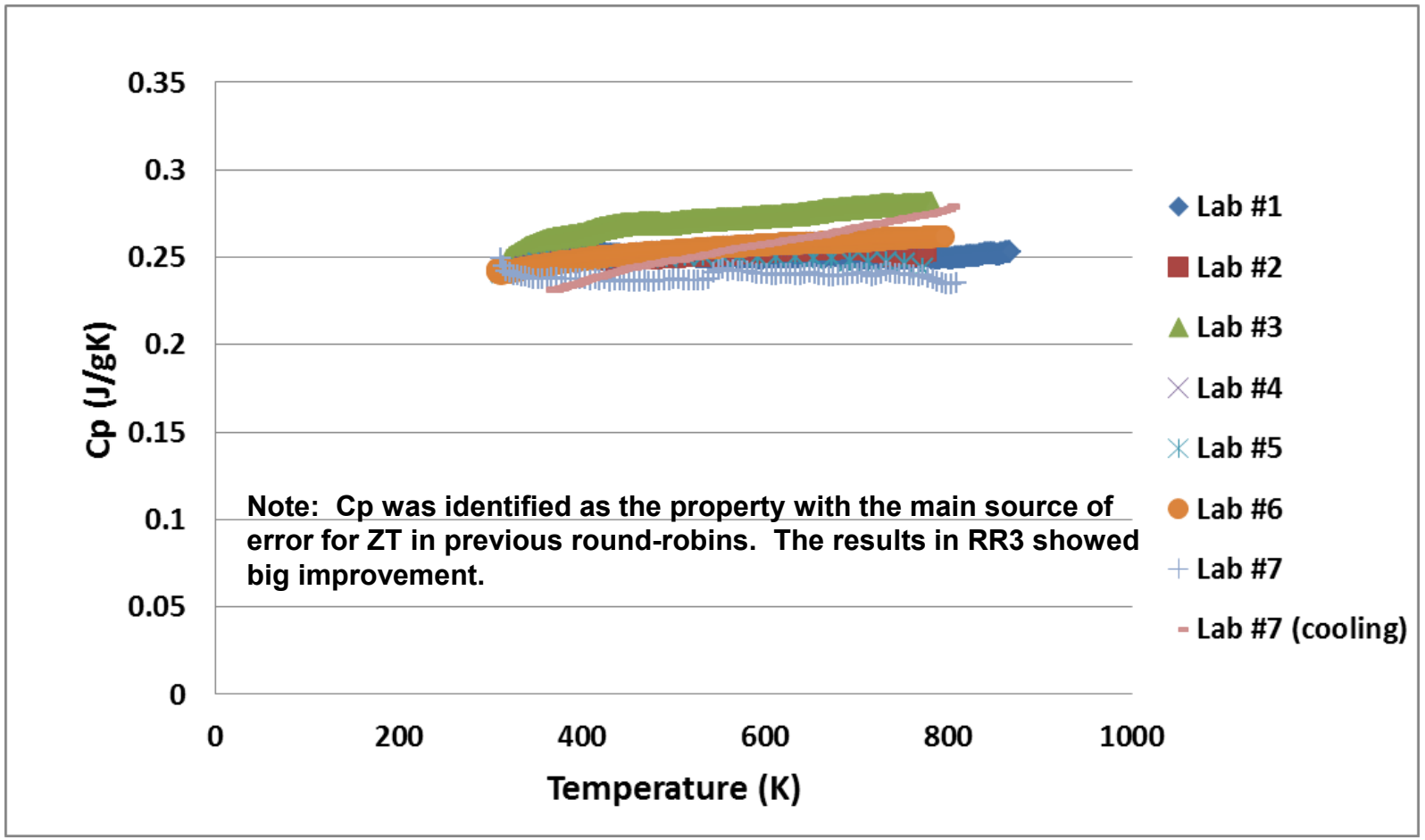
# Technical Accomplishments – 4 of 12

## IEA-AMT RR3 Specific Heat




**Much smaller scatters compare to previous round-robins**



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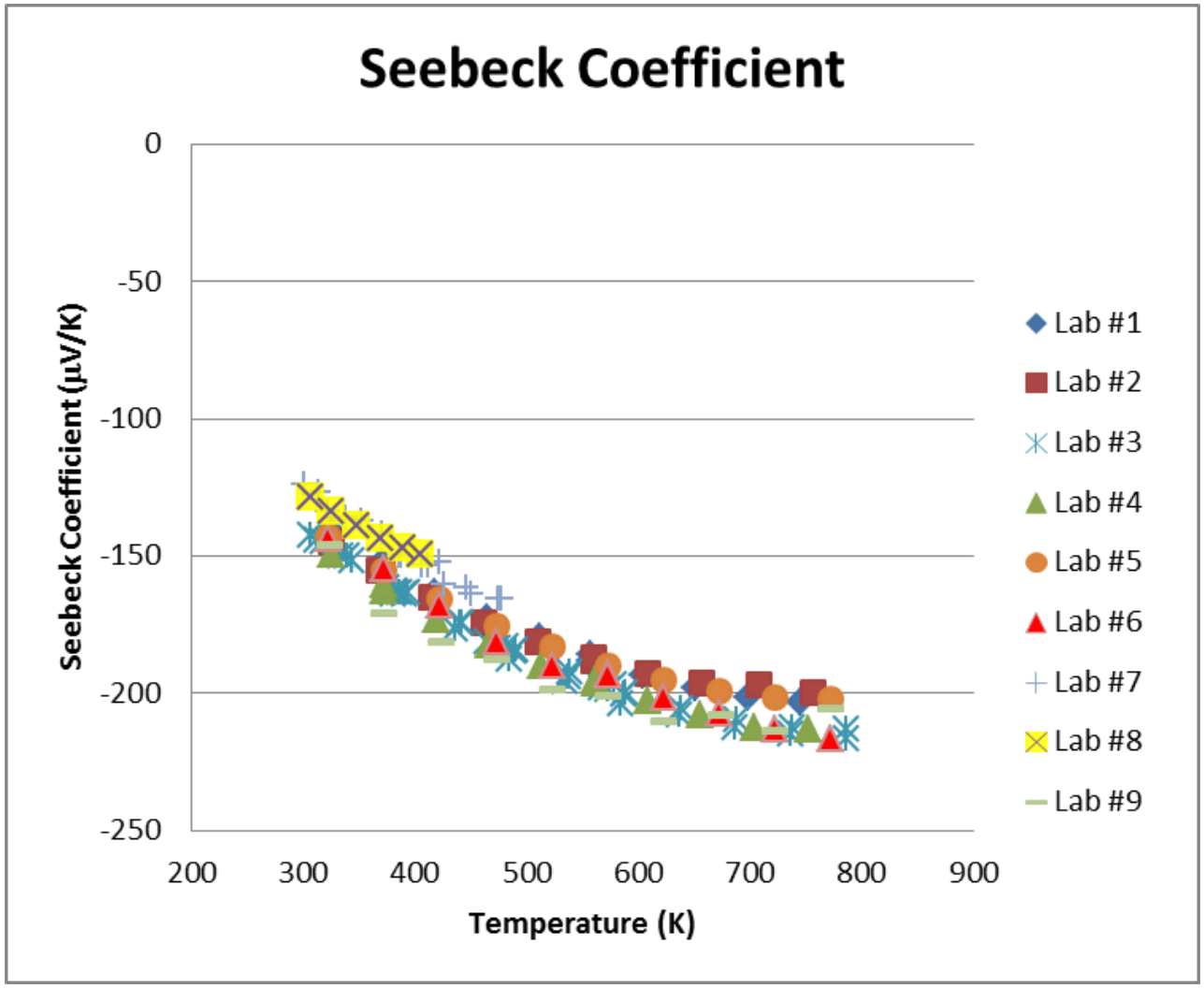
# Technical Accomplishments – 5 of 12

## IEA-AMT RR3 Seebeck Coefficient




**All 4-point measurements had agreement and Two two-point systems were lower**



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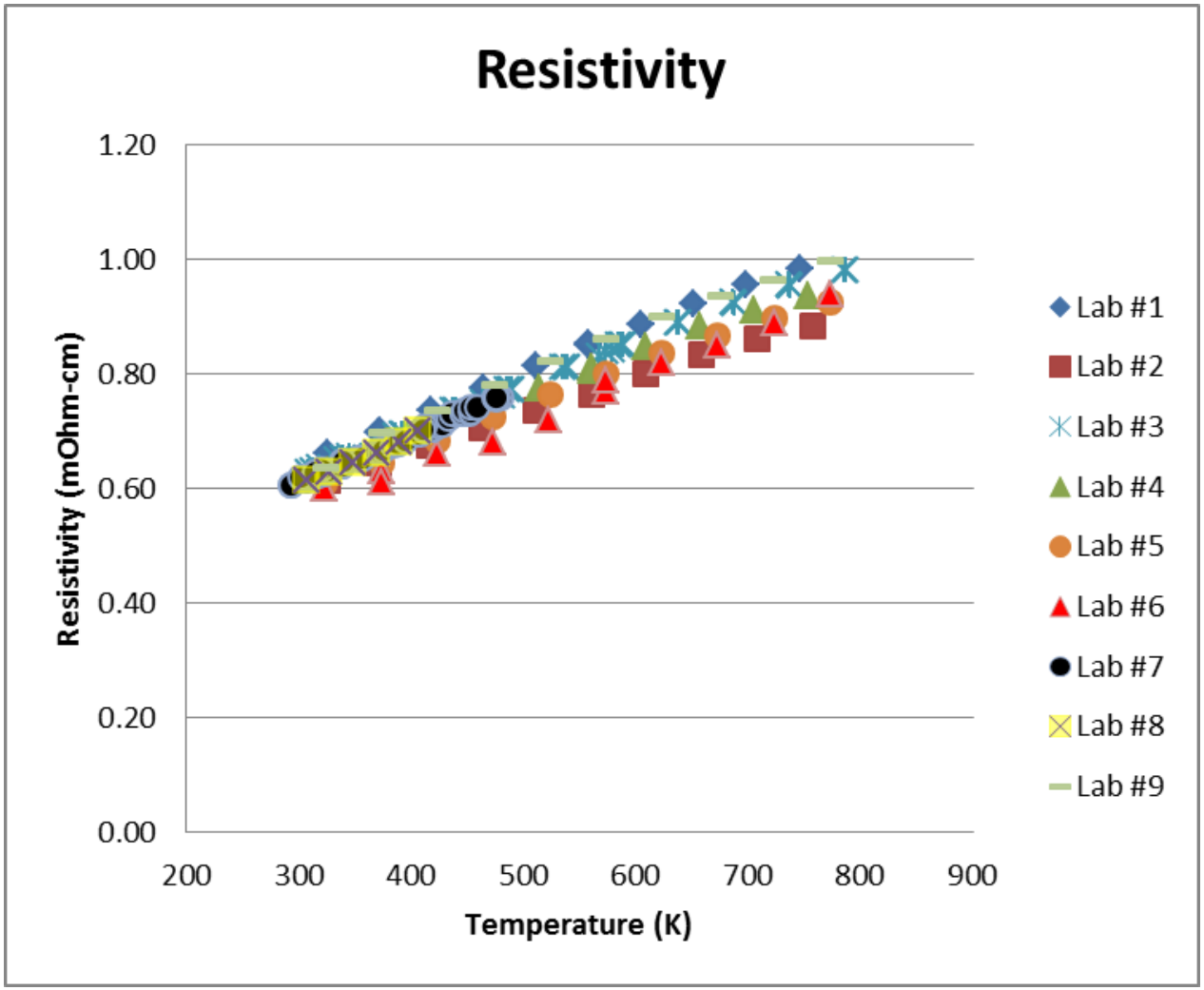
# Technical Accomplishments – 6 of 12

## IEA-AMT RR3: Electrical Resistivity




All 4-point measurements had agreement



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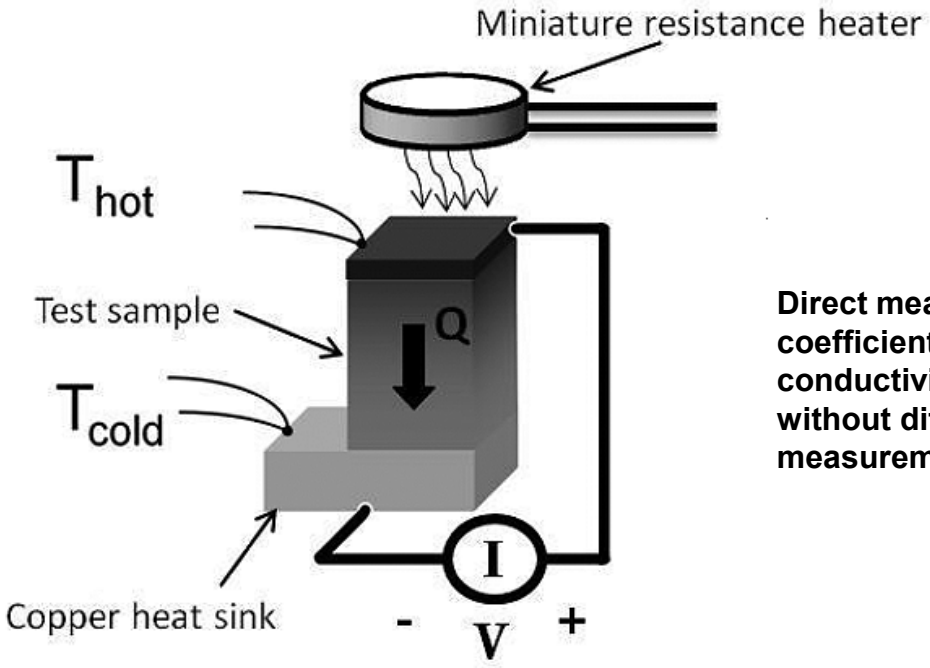
# Technical Accomplishments – 7 of 12

## A sample was tested at ARL using the new steady-state isotherm method

**Alternative Approaches**




**Direct measurements of Seebeck coefficient, resistivity and thermal conductivity. ZT can be obtained without diffusivity and specific heat measurements**

**The sample at ARL was sent to Marlow Industries for the Harman Test**



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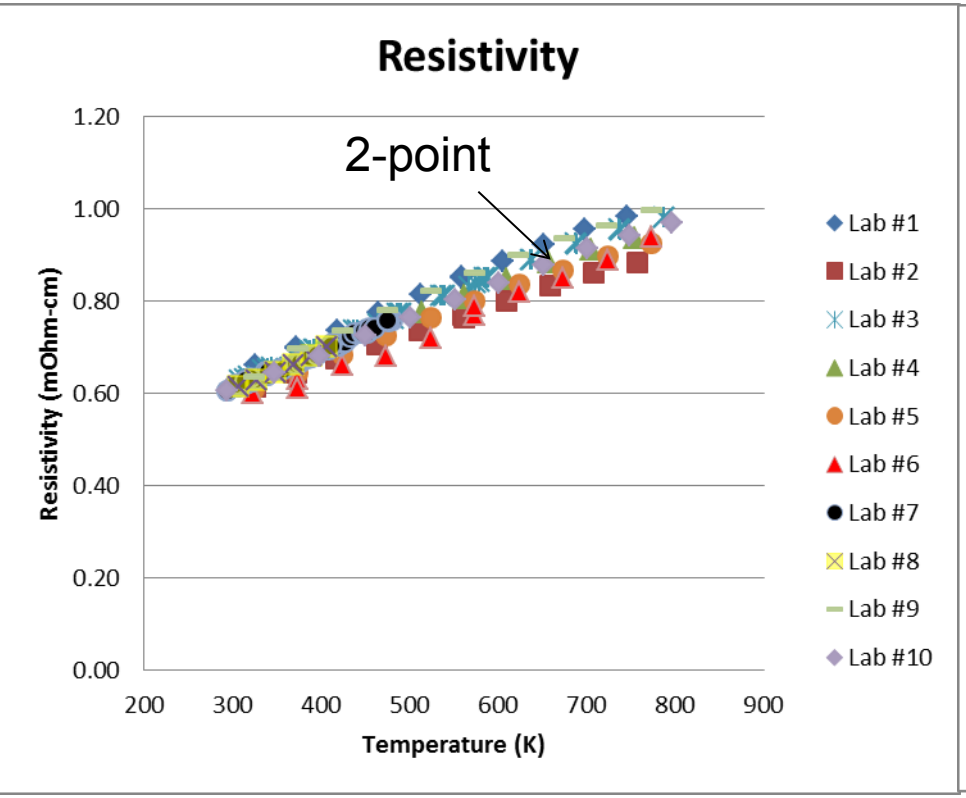
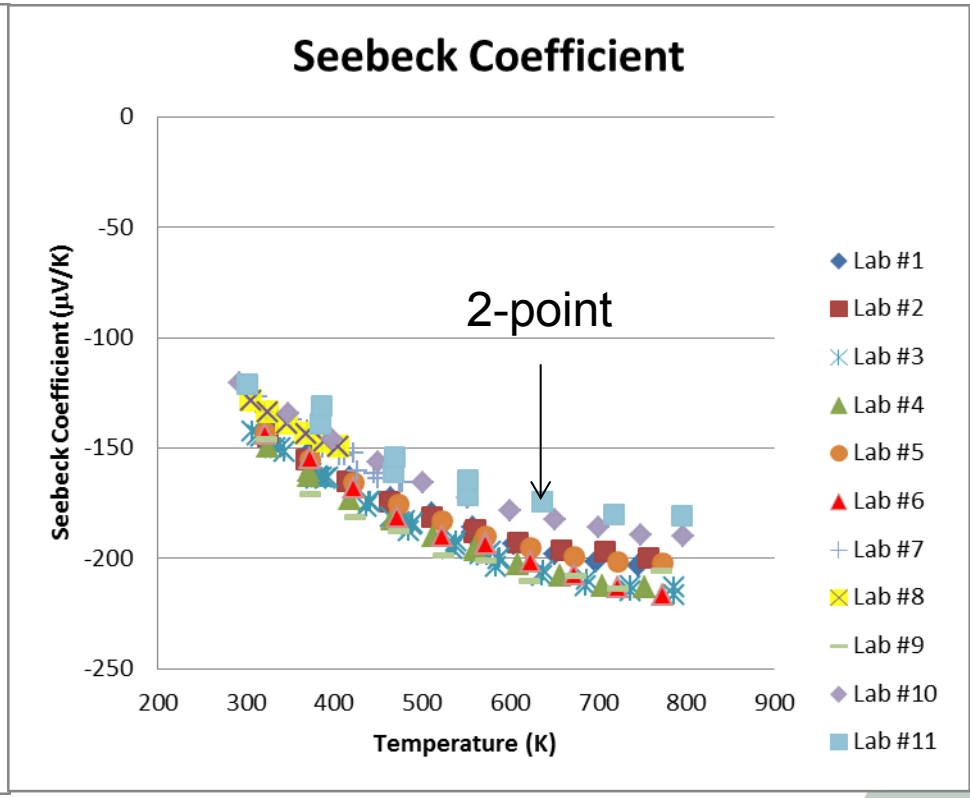
# Technical Accomplishments – 8 of 12

## Adding NIST Results (2-point tests were indicated)




4-point vs. 2-point tests
Perfect match in resistivity
Clear difference in Seebeck coefficient



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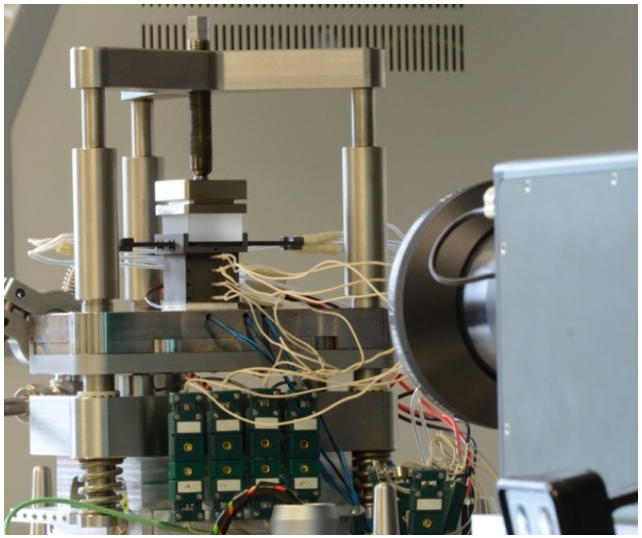
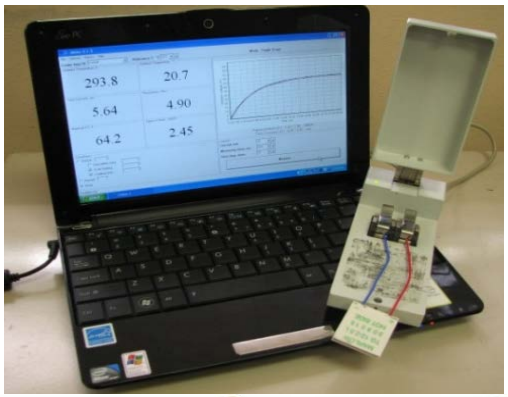
# Technical Accomplishments – 9 of 12

## Survey of Module Efficiency Testing Systems

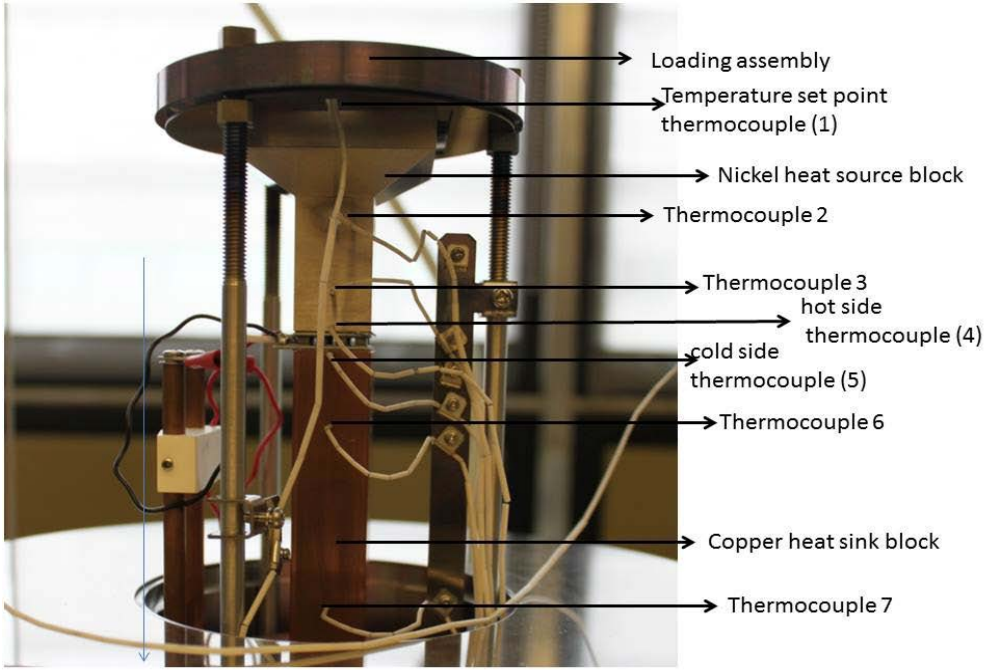
Japan AIST



Germany Fraunhoher IMP




USA
Harman Tester
Marlow and ORNL




GM Global R&D
ULVAC-Riko PEM-3



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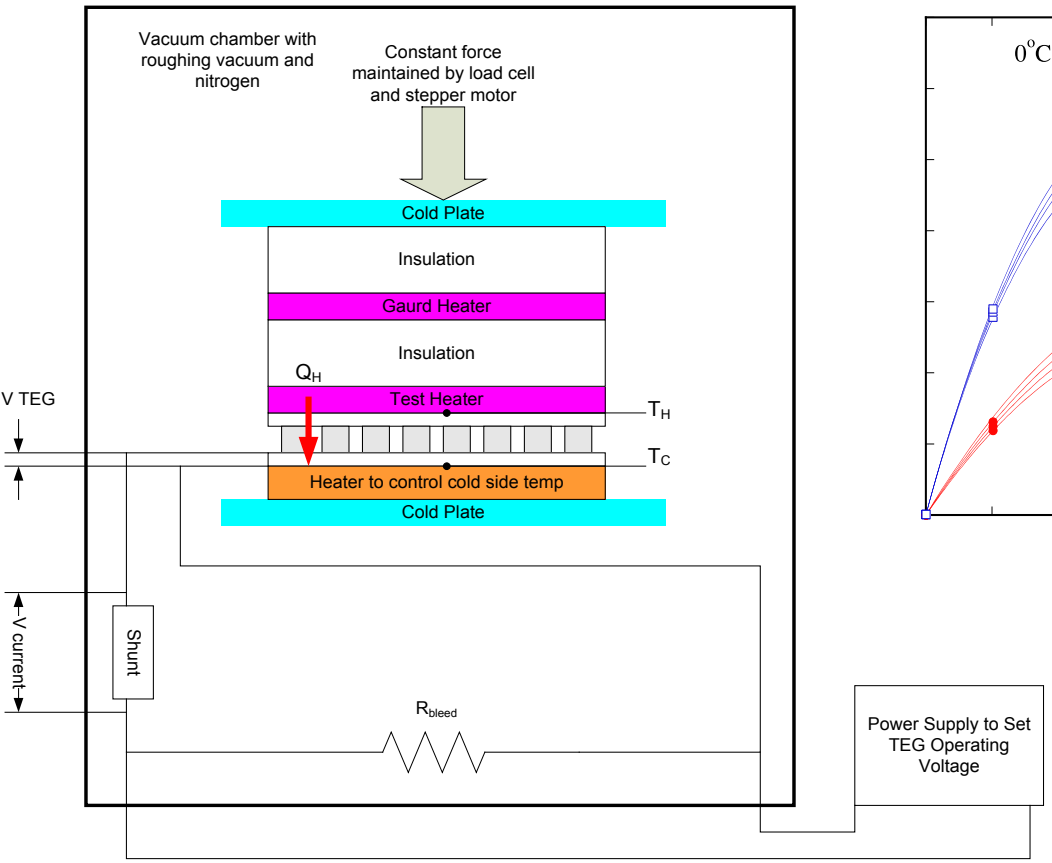
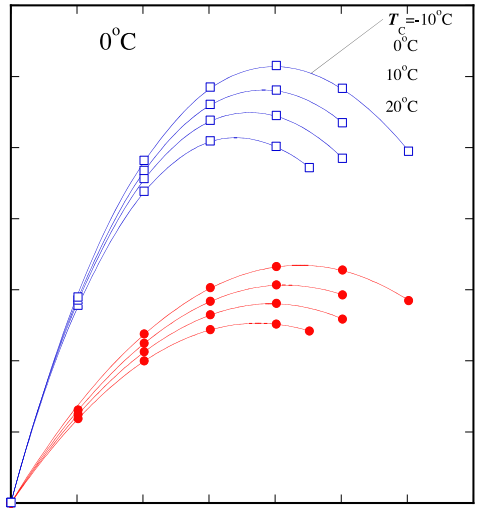
# Technical Accomplishments – 10 of 12

## Survey of Module Efficiency Testing Systems

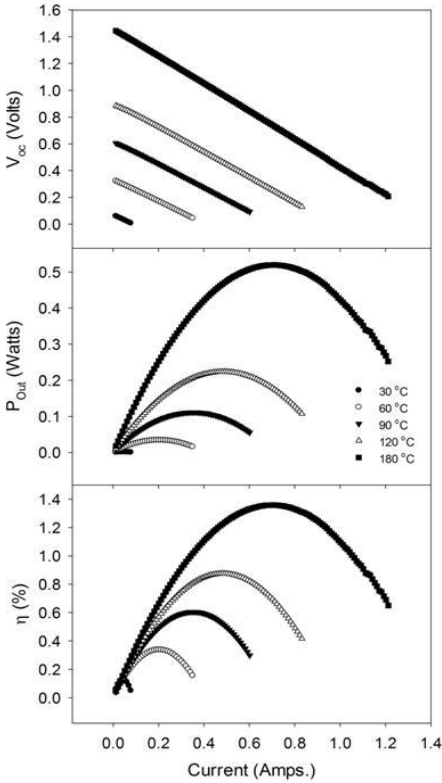
### Marlow Efficiency Test Stand




### AIST Efficiency Results




### GM Efficiency Results



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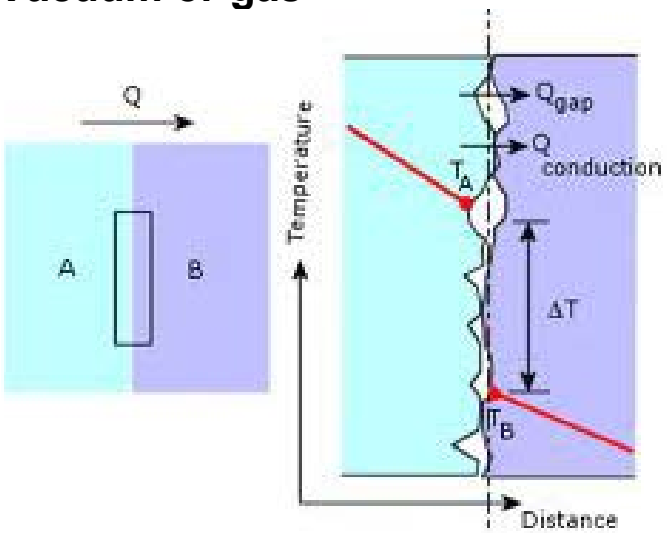
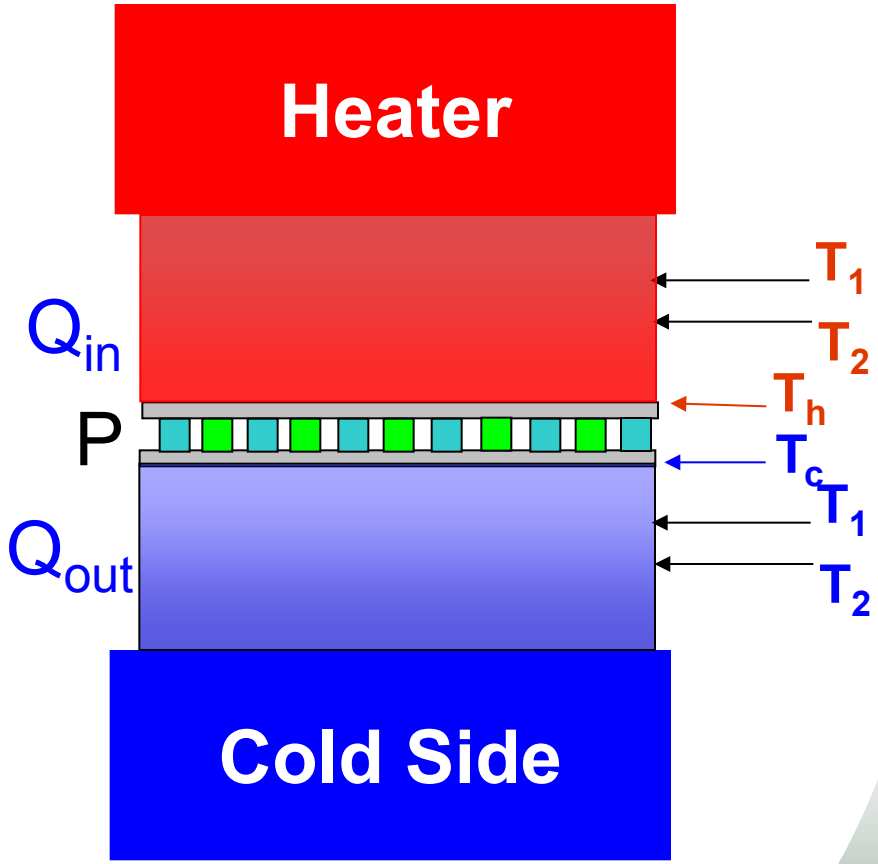
# Technical Accomplishments – 11 of 12

## Important Factors Affecting Efficiency Tests

- **Measurements of $Q_{in}$ and/or $Q_{out}$ (heat loss)**
  - **Radiation loss: ~$T^4$**
  - **Estimating Q from large $\Delta T$ and thermal conductivity**
  - **Convection heat loss: vacuum or gas**
- **Interface resistance:**
  - **Constant test pressure**
  - **Consistent interface**
  - **Vacuum or gas**




TEG Test



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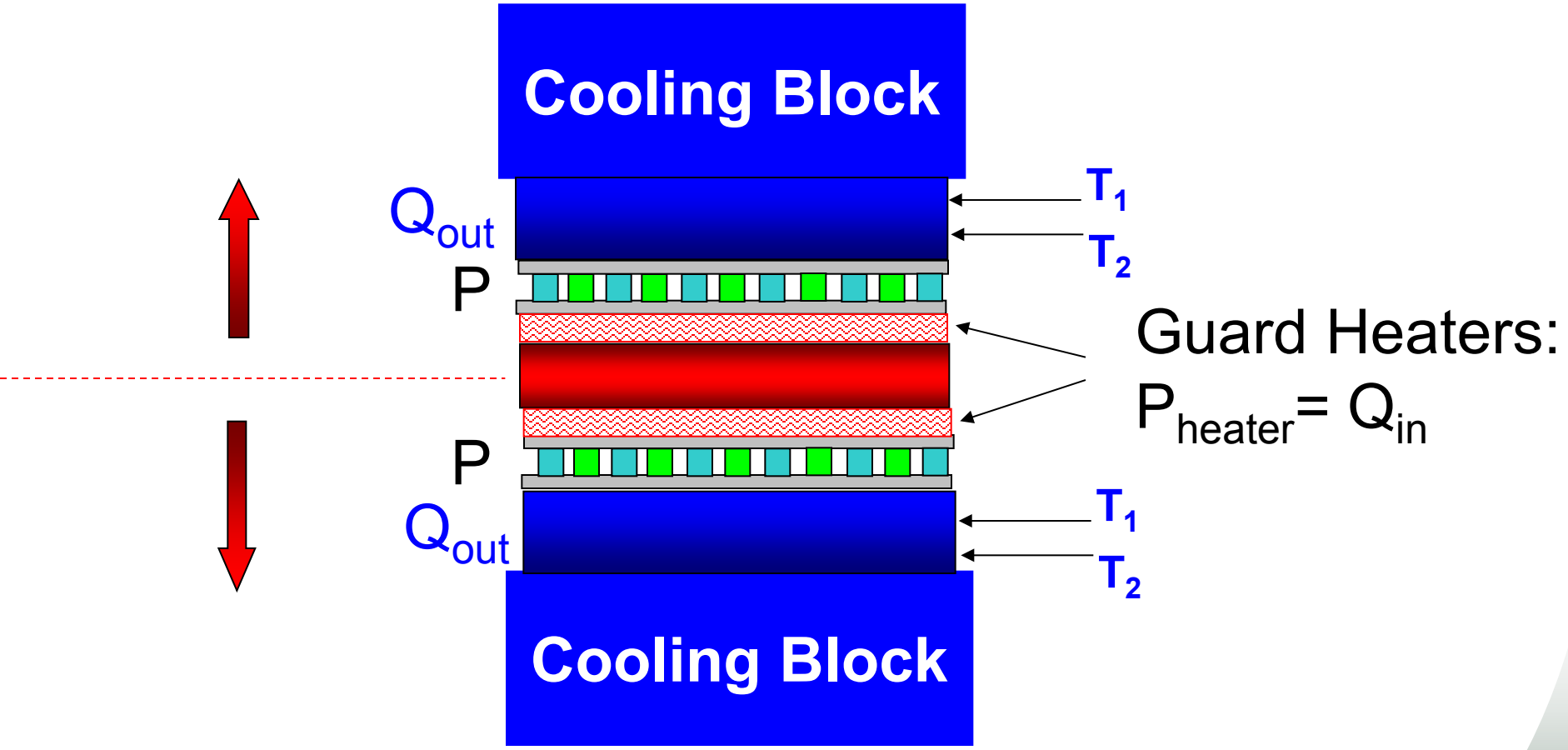
# Technical Accomplishments – 12 of 12

## A Proposed Ideal Testing Setup




Totally Symmetric Test Stand



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# Collaboration and Coordination with Other Institutions

**This project is a collaboration among many countries and research laboratories. ORNL is the leading institute and coordinator**

***Bulk Materials Round Round-Robin 3:***

- **USA: GMZ (G. Joshi); Clemson (T. Tritt); Marlow (J. Sharp); GM R&D (J. Salvador); Army Research Lab (P. Taylor), NIST (J. Martin)**
- **China: SICCAS (S.Q. Bai, L. Chen)**
- **Canada: CANMET(Y.C. Tseng); University of Waterloo (H. Kleinke);**
- **Germany: Fraunhofer IPM (J. König)**
- **United Kingdom: NPL (A. Cuenat)**
- **South Korea: KERI (M.H. Lee)**

***Efficiency Testing Survey:***

- **USA: Marlow (J. Sharp); GM R&D (J. Salvador)**
- **China: SICCAS (S.Q. Bai, L. Chen)**
- **Germany: Fraunhofer IPM (J. König)**
- **United Kingdom: NPL (A. Cuenat)**
- **Japan: AIST (A. Yamamoto)**



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# Remaining Challenges and Barriers

- Additional materials issues that were not addressed may become important such as interconnect materials, interface materials, mechanical reliability and long term stability
- Device and system performance testing may vary from system to system and may be hard to define
- Lack of standards and testing protocols may slow down the deployment of the system to mass production vehicles



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# Future Work

## FY2015 plans

- Finalize the IEA-AMT test procedures for transport properties of bulk materials and 2nd topical report to IEA
- Collaborate with international partners to establish international test standards, e.g. ISO standard
- Complete the set up and initial testing of heat flow-based module efficiency test stand at ORNL
- Prepare to conclude IEA-AMT Annex VIII and propose to organize a new annex on device performance testing



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# Summary

- The current project, through IEA-AMT Annex VIII, is addressing the important issue of measurement standardization of thermoelectrics
- Significant improvement in materials properties measurements results were obtained in round-robin 3, especially in specific heat
- Standard test procedures have been developed and modified after the round-robins
- Initial survey of module efficiency testing has been conducted
- The project continues to focus on the critical issues during the transition from materials R&D to commercialization



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